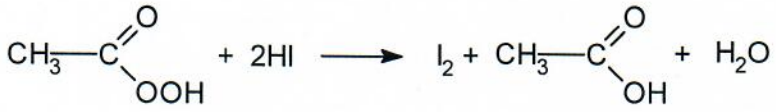
ENFORCEMENT ANALYTICAL METHOD (OPPTS 830.1800) FOR THE ANALYSIS OF THE PAA AND HYDROGEN PEROXIDE ACTIVE INGREDIENTS IN REDUCX

CHEMICAL BACKGROUND

The hydrogen peroxide content is determined by an oxidation-reduction titration with ceric sulfate, according to equation 1. After the endpoint of this titration has been reached, an excess of potassium iodide is added to the solution. The hydriodic acid formed in acidic media reacts with peracetic acid to liberate iodine, according to equations 2. A standard solution of sodium thiosulfate is used to titrate the liberated iodine, as shown in equation 3. The endpoint of this titration is used to calculate the peracetic acid content.

1. $H_2O_2 + 2\ Ce(SO_4)_2 \rightarrow Ce_2(SO_4)_3 + H_2SO_4 + O_2$

2. $2\ KI + H_2SO_4 \rightarrow 2\ HI + K_2SO_4$



3. $I_2 + 2\ S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2\ I^-$

The time needed to complete the analytical procedure including the following determinative step is approximately 10 minutes per sample.

The ceric sulfate-sodium thiosulfate titration is the classic technique used to measure hydrogen peroxide and peracetic acid in an equilibrium solution such as ReducX. Although copies of the Performing Laboratory's Standard Operating Procedures are on the following pages, the method has been accepted by EPA and was used in a previously submitted and accepted study (MRID 46067001).

ENVIRO TECH CHEMICAL SERVICES STANDARD OPERATING PROCEDURE

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Without a yellow control label to the right of this statement, this procedure is a draft. A draft or an uncontrolled copy cannot be used to manage a process or a task.			
Revised Section(s): Previous revision expired			

I. TITLE: DETERMINATION OF HYDROGEN PEROXIDE AND PERACETIC ACID IN SOLUTIONS

II. PURPOSE: This document is to be used by any lab personnel involved in the analysis of the active ingredients hydrogen peroxide and peracetic acid (PAA) in ETCS concentrated solutions.

III. EQUIPMENT:

665 Dosimat Digital Titrator or equivalent (to dispense 0.1 N Ceric Sulfate)
Calibrated scale capable of reading to at least 0.001g
120 ml sample beaker/cup
Disposable transfer pipettes
50 ml graduated cylinder
Magnetic stirrer and stir bars
Digital burette capable of dispensing in single instruments of 0.01 ml with accuracy $\pm$0.2% or equivalent (to dispense 0.1 N Sodium Thiosulfate)

IV. REAGENTS:

Ceric Sulfate, 0.1N
0.5% Starch indicator solution
Ferroin Indicator
Potassium iodide crystals, ACS
0.9 N Sulfuric Acid, cooled
0.1 N Sodium Thiosulfate, $Na_2S_2O_3$
De-ionized or reverse osmosis water
De-ionized or reverse osmosis ice cubes/chips. (One cube/chip should weigh around 7 g)

V. PROCEDURE:

1. Weigh out 1.5-2 grams of PAA sample and use DI or RO water to dilute the sample 10 times. (Ex. 4 g PAA x 10=finished wt. of 40 g; therefore, if your sample weight is 4 g then add 36 g of RO water to bring the finished wt. to 40 g) Mix well, cover, and set aside. Hint: The 10-1 PAA dilution expires15-20 min. after being made.

2. Take a 120 ml sample beaker/cup and place a RO ice cube inside it.

3. Using a 50 ml graduated cylinder, measure out approximately 50 ml of 0.9 N sulfuric acid, and then pour it into the sample cup.

4. Place the sample cup on the balance and press tare.

5. Take a disposable transfer pipette and use it to weigh out approximately 1.0-2.0 grams of your diluted PAA sample. Record the exact weight.

6. Place a stir bar in the sample cup and place it on a magnetic stirrer base and turn stirrer on to a slow/medium speed. Insert the dispensing tip of the digital titrator into the sample cup so that the tip is completely submerged.

7. Add 7-9 drops of ferroin indicator and then begin manually titrating the sample with 0.9 N Ceric Sulfate. Titrate at a moderate speed until the orange color begins to fade and a purple color starts appearing. Slow the Ceric dispensing speed down so you can see the color gradually change from purple to pale blue. Hint: A blue/green color is past the end point. If the sample color indicates that it is past the endpoint, **do not continue**. Discard the sample and try again.

8. Remove the dispenser tip and add 1 scoop of Potassium Iodide crystals.

9. Begin titrating with 0.1 N Sodium Thiosulfate. The sample will start a dark brown and will change to an orange/salmon color. While some brown coloring is still visible add approximately 2 mL of 0.5% starch indicator solution. The sample will darken again. Begin dispensing one drop at a time until the sample turns bright orange and holds the color for 10-15 sec. Hint: 5% PAA uses around 2 ml and 15% uses around 5 ml, depending on the sample weight.

10. Record the exact amount of 0.1 N Sodium Thiosulfate used.

11. Record the exact amount of 0.1 N Ceric Sulfate used.
12. Calculate the % H_2O_2 and % PAA by plugging your recorded amounts in the formulas listed below.

13. Repeat this procedure until the H_2O_2 and PAA results are repeatable and the replications calculate to be within 0.08% of each other.

FORMULAS:

$$\frac{(\text{ml 0.1 N Ceric Sulfate} \times 0.17)}{\text{g sample}} \times 10 = \%\ H_2O_2$$

$$\frac{(\text{ml 0.1 N Sodium Thiosulfate} \times 0.38)}{\text{g sample}} \times 10 = \%\ \text{PAA}$$